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Insufficient Reporting of X-ray Photoelectron Spectroscopy Instrumental and Peak Fitting Parameters (Metadata) in the Scientific Literature

Running title: Insufficient Reporting of XPS Parameters

Running Authors: Major et al.

George H. Major,[1] B. Maxwell Clark,[1] Kevin Cayabyab,[1] Nathan Engel,[1] Christopher D. Easton,[2] Jan Čechal,[3] Donald R. Baer,[4] Jeff Terry,[5] Matthew R. Linford[1,a)]

[1]Department of Chemistry and Biochemistry, Brigham Young University, Provo UT 84062, USA
[2]CSIRO Manufacturing, Ian Wark Laboratories, Clayton, Victoria 3168, Australia
[3]CEITEC BUT, Brno University of Technology, Purkyňova 123, 612 00, Brno, Czech Republic
[4]Pacific Northwest National Laboratory, Richland WA, 99354, USA
[5]Department of Physics, llinois Institute of Technology, 3101 S. Dearborn St., Chicago, Illinois 60616, USA

[a)]Electronic mail: mrlinford@chem.byu.edu

This study was motivated by earlier observations. It is a systematic examination of the adequacy of reporting of information (metadata) necessary to understand X-ray photoelectron spectroscopy (XPS) data collection and data analysis in the scientific literature. The information for this study was obtained from papers published in three high-quality journals over a six-month period in 2019 and throughout 2021. Each paper was evaluated to determine whether the authors had reported (percentages of the papers properly providing the information are given in parentheses) the spectrometer (66%), fitting software (15%), X-ray source (40%), pass energy (10%), spot size (5%), synthetic peak shapes in fits (10%), backgrounds in fits (10%), whether the XPS data is shown in the main body of the paper or in the supporting information (or both), and whether fitted or unfitted spectra were shown (80% of published spectra are fit). The Shirley background is the most widely used background in XPS peak fitting. The Al Kα source is the most widely used X-ray source for XPS data collection. CasaXPS is the most widely used fitting program for XPS data analysis. There is good agreement between the results gathered during the two years of our survey. There are some hints the situation may be improving. This study also provides a list of the information/parameters that should be reported when XPS is performed.

I. INTRODUCTION

We report a survey of reported X-ray photoelectron spectroscopy (XPS) metadata with a focus on parameters associated with data collection and peak fitting. The outcome of this study highlights inadequacies in reporting that contribute to the propagation of incorrect analyses appearing in the scientific literature. Building from this survey, a list of important parameters is provided to assist newer users in recording and reporting XPS data in a manner that will be useful and informative to the research community.

XPS is a surface analysis method that is based on the photoelectric effect. XPS identifies the elements and the chemical states of the elements at surfaces. The use of XPS has increased dramatically over the last four decades to the point that it is now a well-established technique.[1] XPS continues to bring in new users and widen its scope of influence in other fields.[2-6] The increasing demand for XPS coincides with growing applications of thin films and nanomaterials and a recognition of the importance of the chemical species present at surfaces in many areas of science and technology.[7] XPS is now available in multiple modes, including angle resolved, operando, near ambient pressure (NAP), imaging, large-area analysis, in conjunction with depth profiling, and hard XPS (HAXPES).[8] Conventional XPS is sensitive to the outermost 5 – 10 nm of materials.[9] This surface sensitivity is a result of the limited distance photoelectrons can travel in materials without losing energy, not the depth that X-rays penetrate into materials, which is much greater.[10, 11] Electrons that have lost energy contribute to the background around photoelectron peaks.[12] The fitting of XPS backgrounds is important for determining peak intensities, i.e., for separating the so-called zero-loss signals from the background of inelastically scattered electrons.[13] Analysis of XPS backgrounds can also provide information about the layers/thin films or nanostructure at a surface.[14, 15]

Modern XPS instruments collect data that is usually precise, of high quality, and reproducible. XPS can also be accurate when samples are homogeneous and standards are appropriately applied. However, many samples are inhomogeneous in depth or laterally. Accordingly, the assumption that the composition of an analysis volume is uniform, which is often made in XPS quantitation, is not always true. XPS spectra are further complicated by the presence of multiple peaks from a given element, overlapping signals from multiple elements, chemically shifted peaks, spin-orbit splitting, shake-up and shake-off signals, plasmon signals, multiplet signals, Auger peaks, peak asymmetry, the valence band region, and rising backgrounds/baselines. For example, the presence of shake signals can make quantification challenging.[16, 17] Furthermore, while some XPS baselines are simple, others, e.g., from open shell metals, are extremely complicated.[18-21] With proper interpretation, the diversity of spectral features in XPS increases the wealth and quality of information available from the technique. These complications also make the reporting of the methods used to collect and analyze XPS data critically important.

As the use of XPS has expanded, XPS experts have increasingly observed problems with some of the XPS data, data analysis, and reporting of experimental conditions and parameters in the scientific literature.[22] Incorrect data analysis can be misleading to non-experts. It may also be propagated in future studies.[23-25] Some of us recently participated in a study that examined some of the XPS data analysis in the literature. We observed that ca. 40% of the peak fitted XPS narrow scans reported over a six-month period in three well-respected journals in 2019 were incorrect.[22, 26] Unfortunately, this problem is not unique to XPS. Experts in other surface and material analytical techniques similarly suggest that 20-30% of the data analysis related to these methods is flawed.[27, 28] Having been aware of this issue for decades, and even anticipating it, the XPS community has produced standards, e.g., from ASTM E42 on surface analysis and from ISO TC201 on Surface Chemical Analysis, books, book chapter, videos, reference spectra, and other tutorial documents to try to educate the technical community. A recent effort along these lines was a series of guides in the Journal of Vacuum Science and Technology written by XPS experts.[29] They cover many aspects of the technique, including experiment planning,[7] sample handling,[30] instrument calibration,[31] charge referencing,[32] probing depth,[9, 33] lateral resolution,[34] peak fitting,[35] quantitative analysis,[17, 36] analysis of nanoparticles,[37] epitaxial films,[38] the carbon 1s region,[39] catalytic materials,[40] and analysis of polymers.[41]

This paper is an expansion of our previous study that assessed the quality of XPS data and peak fitting.[22, 26] It examines the reporting of key parameters and information related to XPS data acquisition and peak fitting. These parameters are important. Insufficient reporting of instrumental and analysis parameters limits the ability of other scientists to assess the correctness of results and replicate them.[42, 43] In this work, we focus on some of the basic parameters that should be reported. In her talk at AVS in 2019, Karen Gaskill also discussed and presented data showing the lack of reporting of parameters related to XPS data collection.[42, 43] At the end of this paper, we provide recommendations regarding which data acquisition and analysis parameters should be reported. The results in this paper were derived from a survey of the XPS data and related reporting in over 1000 papers published over two periods of time: six months of 2019 and all of 2021. These papers were published in three well-respected journals from established publishers.

II. METHODOLOGY AND DATA MINING

The XPS data evaluated in this study were originally published in three high-quality journals from respected publishers over two time periods: from January to July of 2019 and in all of 2021. These journals, which we refer to as Journals A, B, and C, were chosen because we believe their papers are representative of how XPS is currently used. These journals are not identified to avoid associating them with the issues flagged here. Indeed, the issues that are discussed in this paper do not appear to be unique to these journals. XPS experts regularly see the same problems in other journals. Journals A, B, and C had impact factors of 25 – 30, 4 – 4.5, and 4 – 5 in 2019 and 2021, respectively. More specifically, Journal A is an energy/battery materials journal, Journal B focuses on materials and surfaces, and Journal C is a general science journal. From 2019 and 2021, we examined 152 and 346 papers in Journal A, 152 and 286 papers in Journal B, and 104 and 265 papers in Journal C, respectively. The papers from 2019 were the same used in our previous study.[26] Additional papers from 2021 were considered because it was felt that those from 2019 alone were inadequate to fully evaluate the parameters under consideration. It was also unclear whether the same problems in the literature would be present at two different times. As shown below, both years yielded very similar results. Publications containing XPS results in Journals A, B, and C were identified using a keyword search on 'XPS.' However, only papers that claimed to have performed XPS were included in the survey. If the paper only referenced XPS data done from a different study, it was excluded. The following bits of information were mined from each publication:

1. Whether raw data, or only synthetic peaks, were shown.
2. Whether the XPS data were fit.
3. Whether backgrounds were shown under the XPS fits.
4. The synthetic peak shape(s) used in any fits.
5. The background(s) used in any fits.
6. Whether the XPS data were in the main text or supplemental information.
7. The spectrometer used.
8. The XPS fitting software used.
9. The pass energy used for data acquisition.
10. The X-ray source used.
11. The spot size used.

In some cases, it was reasonably clear what these parameters were, such as which X-ray source was used when Auger peaks that are consistent with a particular source were present in a spectrum, or when a Shirley background was used. Nevertheless, only parameters that were explicitly listed in the text, whether in the main body of the paper or in the supplemental information, were included in the tallies. The most challenging category to evaluate was whether the raw data were shown. In most cases, real data could be identified because of noise on it. However, when the data looked artificially smooth or it appeared that only the sum of the synthetic peaks (fit components) was shown, these results were included in the category of not showing the raw data. Admittedly, some very low noise spectra may have been inadvertently included in this category. In a few cases, closely related parameters were grouped into a single category. For example, the different types of synthetic peaks that can be made by summing or multiplying a Gaussian and a Lorentzian[44-46] were placed in a single ‘Gaussian-Lorentzian’ category.

III. RESULTS AND DISCUSSION

A. *Reporting the type of instrument used*

Different commercial XPS instruments have various geometries, measurement modes, and capabilities, which can influence data acquisition and quality. Accordingly, to avoid confusion, the make, model, and manufacturer of the XPS instrument used in a study should be reported, along with any special modes employed. In addition, any non-standard, optional, or modified instrumental features, accessories, or sources, e.g., X-ray or sputtering, should be reported. The following is an example of where confusion may occur without appropriate XPS reporting. The NAP-XPS O 1s spectrum of liquid water shows a narrow O 1s peak at higher binding energy from gas phase water and a broader O 1s peak at lower binding energy from liquid phase water.[47] This result may be puzzling to some readers if it is not clear that an NAP-XPS experiment had been performed. In the three journals we surveyed, ca. one-third of the papers from 2019 and 2021 (34% and 29%, respectively, see Fig. 1) did not report the type of spectrometer used. It is not clear whether the improved reporting in 2021 indicates an actual improvement. It may simply reflect the uncertainty in our measurement. In addition, some of the reporting in the ca. two-thirds of the papers that listed the type of spectrometer employed was incomplete. For example, even papers that reported the manufacturer of the instrument did not always fully report the capabilities or modes of it that were used. In some cases, authors listed the class of an instrument without specifying the specific model that was employed.

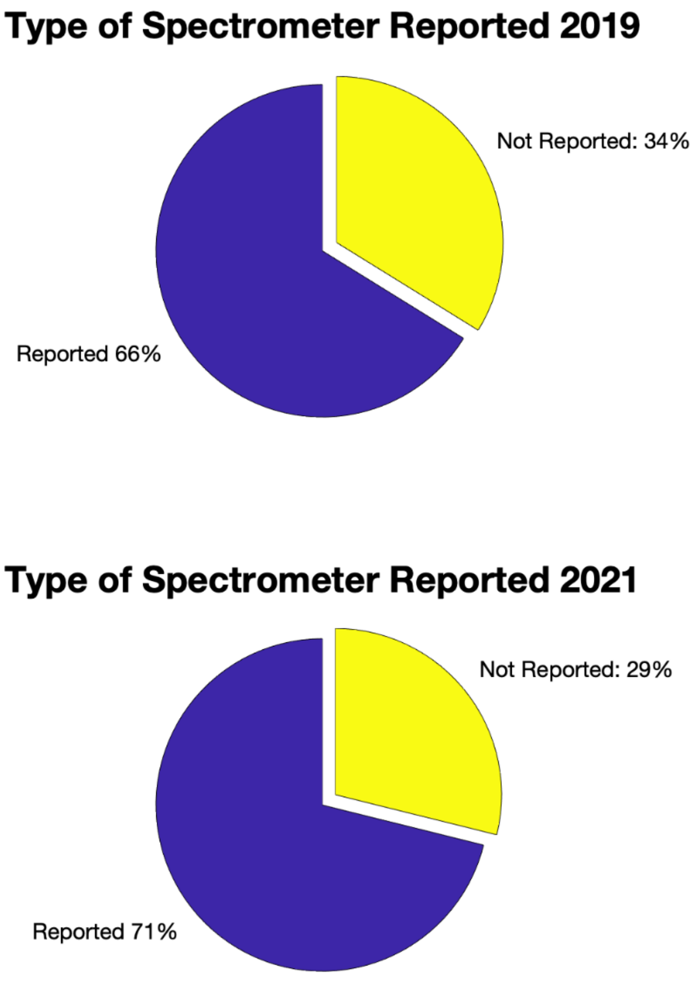

FIG 1. The degree of reporting of the type of XPS instrument used for data acquisition in 408 publications from 2019 and 901 publications from 2021 in Journals A, B, and C.

B. Reporting the X-ray source, pass energy, and spot size

There are important reasons for identifying the X-ray source when XPS is reported. In both 2019 and 2021, only ca. 45% of all the publications in our survey reported the X-ray source that was used, e.g., Al Kα or Mg Kα. (Fig. 2). XPS spectra are most often plotted as a function of binding energy so that photoemission peaks obtained with different X-ray sources will appear at the same positions. However, Auger emission is independent of the energy of the X-ray (excitation) source. When plotted on a binding energy scale, Auger peak positions depend on the X-ray source used. In some cases, choosing a different X-ray source allows Auger peaks to be shifted so that they do not overlap with photoemission lines. The irony here is that the kinetic energies of the photoemission peaks change when the source is changed, not the Auger peaks. In addition, Al Kα X-rays are usually monochromated, while Mg Kα X-rays are not. Monochromated Al Kα X-rays have a narrower line width/energy range (ca. 0.25 eV) than non-monochromated Mg Kα X-rays (ca. 0.7 eV).[8] Thus, peaks generated from monochromatic Al Kα sources are often noticeably narrower. Sometimes, analysts rely on the instrumental details in the Experimental of a paper to determine whether broad peaks in a spectrum are a result of non-monochromated X-rays, the data were deliberately collected at low resolution, the sample is the source of the broadening, or the data are of poor quality. Satellites are present in XPS spectra collected with non-monochromatic Kα X-rays. They may be misidentified if a reader is unaware that the data were collected with a non-monochromatic source. For example, the Kβ satellite peaks from Mg and Al are shifted by 48 eV and 70 eV, respectively, to the low binding energy side of the main peak with about 3% of its intensity. Bremsstrahlung radiation that is not removed in monochromation leads to higher backgrounds, where these extra counts decrease signal-to-noise ratios of peaks.[48] While the advantages of monochromatic sources outweigh their disadvantages, and most conventional XPS is now done with monochromated Al Kα X-rays, non-monochromatic sources provide certain benefits in XPS. Non-monochromatic Mg Kα X-rays generally pass through a thin metal window that is close to the sample. Photo- and Auger electrons generated from this foil help reduce sample charging. Mg Kα X-ray sources may also be more intense than Al Kα sources because X-rays are not lost in monochromation. Often, Mg Kα X-ray sources are less tightly focused than monochromated Al Kα sources, which aids in charge compensation. Finally, the X-ray energy directly affects the sampling depth of an XPS experiment because it directly affects the kinetic energies of photoelectrons. XPS with Mg Kα X-rays is a little more surface sensitive than with Al Kα X-rays, and XPS with both of these sources is more surface sensitive than with the sources used in HAXPES. For all these reasons, and also because of the new modes and sources of XPS that continue to be introduced (ThermoFisher is offering a dual Zr/Ti anode as an alternative to Al/Mg), it is critical to list the X-ray source when reporting XPS results.

The pass energy can significantly affect the peak widths/resolution in an XPS experiment. Figure 2 shows that the pass energy was only reported in ca. 10% of the papers in our survey of the literature in both 2019 and 2021. In some instruments, the ‘pass energy’ is referred to as the ‘resolution’. There is a relationship between pass energy, count rate, and resolution (peak width) in XPS. Higher pass energies allow more electrons to pass through the spectrometer (more counts), but spectra collected under these conditions generally have lower resolution (contain broader peaks), and vice versa. There is a point of diminishing returns in pass energy reduction. While lowering the pass energy beyond a certain point will not reduce peak widths, it will decrease count rates and thereby lessen signal-to-noise ratios.

The area of analysis, influenced by the X-ray spot size, beam rastering, and analyzer lens parameters, is also important information. Figure 2 shows that only ca. 5% of the XPS papers we surveyed in 2019 and 2021 reported the X-ray spot size. A larger analysis area averages signals and composition. Heterogeneity on a surface may be missed if the X-ray spot is too large. An analysis of a sample at a single spot can provide a misleading picture of a surface. Papers with XPS results should report the X-ray source, the pass energy, the step size, and the spot size used to acquire the data.

While not specifically evaluated in our study, step size, which is the distance in energy between points in a spectrum, should also be reported for XPS measurements. In general, survey scans are taken at higher pass energies and larger step sizes (0.5 – 1 eV), while narrow scans are taken at lower pass energies and smaller step sizes (0.05 – 0.1 eV) for enhanced resolution. In general, it is difficult to explore chemical state changes, as manifested by chemical shifts, when the step size and pass energy for a spectrum are large.

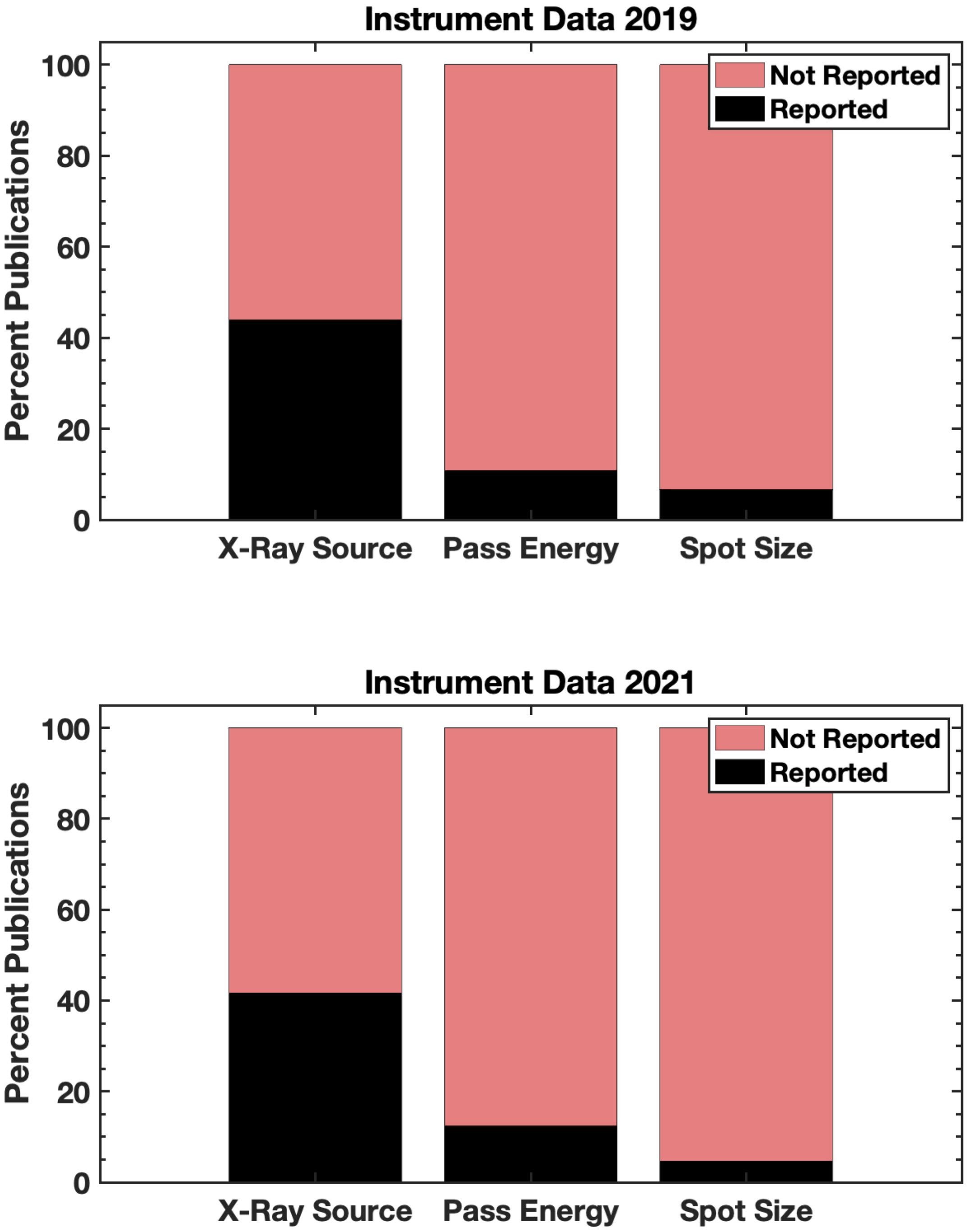

FIG 2. Fraction of publications that reported (black, lower bars) the X-ray source, pass energy, and spot size in 408 XPS-containing publications in 2019 and 901 XPS-containing publications in 2021 in the same three journals.

Figure 3 reveals the XPS publications in 2019 and 2021 that reported the type of X-ray source that was used. Overwhelmingly, XPS practitioners used the Al Kα and Mg Kα sources (98% and 99% of the publications we surveyed in 2019 and 2021 listed one of these two sources, respectively, out of the 44% and 42% of the publications that reported the source that was used). Furthermore, the majority of XPS users employed the Al Kα source, with the fractional use of the Mg Kα source decreasing in 2021. It is not clear whether this decrease in the use of the Mg Kα source is statistically significant. In both years, six publications appear to have mistakenly reported the use of Cu Kα radiation for XPS. All six of these studies used X-ray diffraction (XRD), which often employs Cu Kα radiation. In addition, one paper in 2021 reported the use of a Cr HAXPES source and another the use of a Ag HAXPES source. We expect that HAXPES sources will increasingly be used in the future. In summary, Al Kα and Mg Kα X-rays remain the dominant sources in XPS, with the Al Kα source the most common of the two.

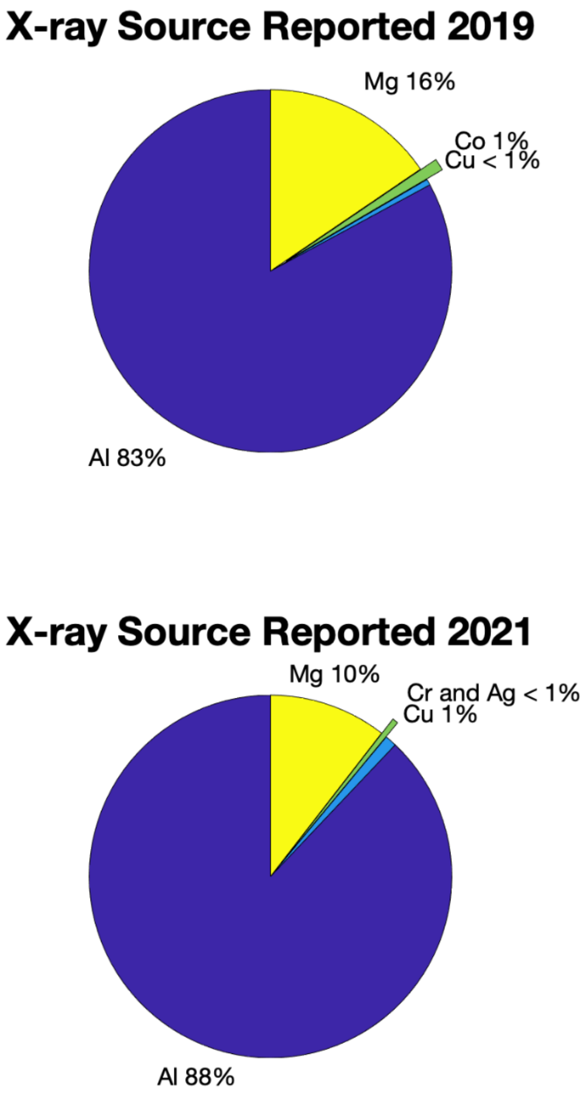

FIG 3. X-ray source reported in 179 papers in 2019 and 374 papers in 2021 from journals A, B, and C. Cr and Ag HAXPES sources were each reported in one publication in 2021. In both years, researchers appear to have mistakenly reported the use of Cu sources, which are used for XRD.

C. Showing and fitting spectra

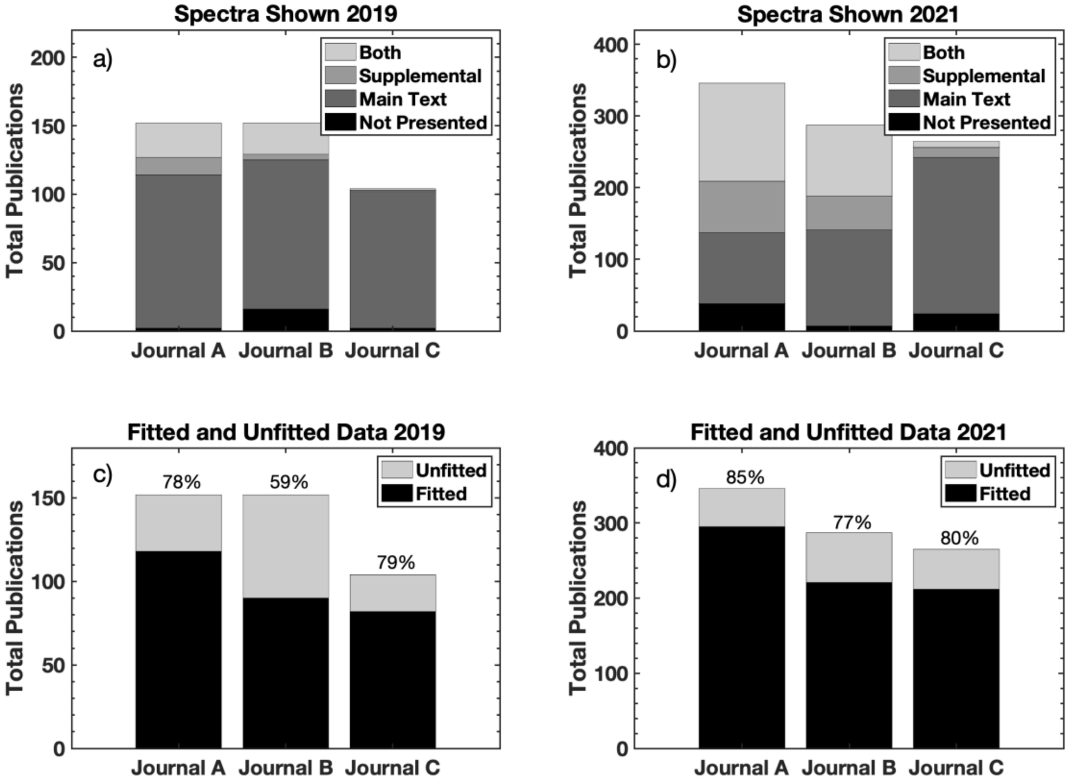

FIG 4. (a) – (b). The locations of the XPS data in Journals A, B, and C in 2019 and 2021. (c) – (d) Fraction of papers with XPS data in Journals A, B, and C in 2019 and 2021 that show peak fitted vs. unfitted XPS spectra. The percentages above the bars in (c) – (d) are the fractions/percentages of the black bars of the total bar height.

The location of the data shown in a paper is often related to its importance. Key and/or representative results are often shown in the main body of a paper, while less important, more specialized, and/or simply large quantities of results that are important but that may not fit in the main body of a paper may be placed in the supporting information. Figure 4 (a) – (b) reveals the locations of the XPS data presented in Journals A, B, and C in (a) 2019 and (b) 2021. In 2019, most of the XPS data in the three journals appeared in the main text. However, in 2021, a larger fraction of the XPS data appeared in the supporting information, especially in Journals A and B. In both 2019 and 2021, most of the XPS spectra in Journal C appeared in the main text of the papers. We strongly support the publication of additional XPS spectra and other experimental findings in the supporting information of papers. A visual presentation of results provides the reader the opportunity to independently evaluate the quality of data and any fits to it, much like including images of blots is important in the literature for biology.[49] As more techniques are used in a study, the amount of supplementary information reported may grow. The use of supporting information can also reflect reviewer and editor preferences. In summary, in a world where publishing is digital, the cost of publishing more information, either in the main text or the supporting information, is low. We encourage the publication of more data and information, especially data that was used to generate quantitative information, such as tables presented in the main text, and/or spectra that were used for qualitative analysis. Not all data needs to or should be published, but in general, more information is always welcome.

Peak fitting is an essential part of much XPS data analysis. Figure 4c – d shows the number of peak-fitted XPS spectra presented anywhere in Journals A, B, and C compared to the total number of spectra (fitted and unfitted) shown in these journals. In all three journals, and in both years of our survey, the fraction of the peak fitted spectra is high. Furthermore, in all cases, the fraction of peak fitted spectra increased in 2021 compared to 2019. It is not clear how statistically significant this trend is, but it is probably something worth watching. It seems safe to conclude that peak fitting remains important and may be becoming more important in XPS data analysis. Also, in both years, some papers reported XPS findings, but did not show any spectra.

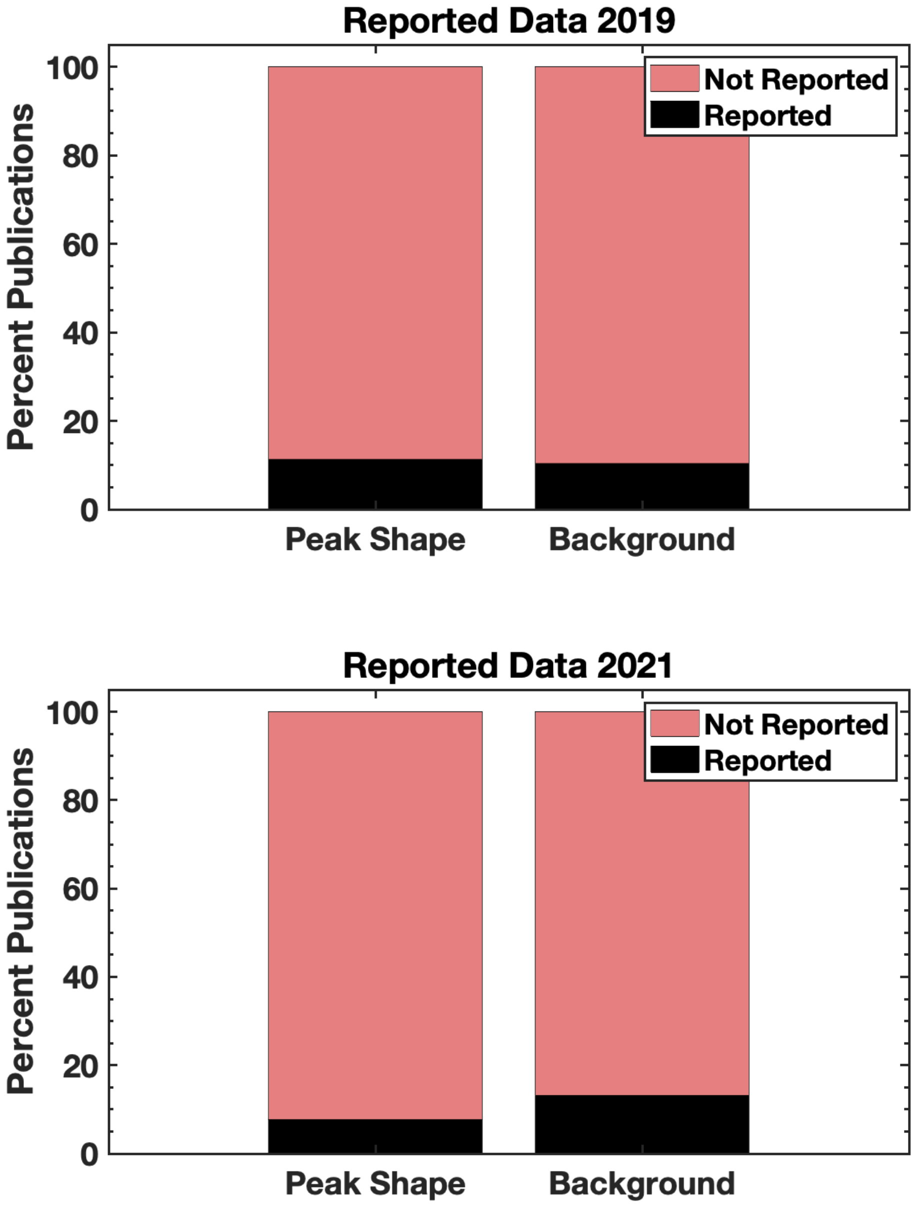

FIG 5. Percentages of publications (black, lower bars) with XPS results from 2019 and 2021 that disclosed the peak shape and background used in peak fitting.

The shapes and positions of the underlying signals/peaks in an XPS peak envelope depend on the specific element and orbital from which photoemission takes place, the sample chemistry, the core-hole lifetime, relaxation physics, the instrument geometry and quality, sample charging, the width of the X-ray source, and the energy resolution (pass energy) of the instrument. For a peak fit, synthetic peaks, which are based on mathematical functions, are chosen to mimic the underlying photoemission signals. The natural line shape of XPS peaks is Lorentzian. However, significant Gaussian character, and even peak tailing, is present in many XPS signals. The most commonly used peak shapes for fitting XPS spectra are the Gaussian-Lorentzian sum (GLS), Gaussian-Lorentzian product (GLP), and Voigt functions, where the Voigt function is a convolution of Gaussian and Lorentzian functions.[46,50] The mathematics of the Voigt function often makes it a better choice for fitting XPS narrow scans. Nevertheless, the GLS and GLP are adequate for many applications.

Various mathematical functions can account for asymmetry in XPS peak fitting.[46, 51] Metallic samples, i.e., materials without bandgaps, often produce asymmetric signals because of promotion of valence electrons into the conduction band of the material during photoemission. The degree of this asymmetry depends on the number of electrons at the Fermi surface. The background of an XPS peak fit represents an attempt to separate the unattenuated signal (conventional photopeak) from counts/signal of inelastically scattered electrons. In some cases, the integrated area of the unattenuated signal depends noticeably on the background placed under it. Most XPS peak fits use one of three backgrounds: the linear, Shirley, or Tougaard-type background.[13] Other more specialized backgrounds are also occasionally employed.[52] The linear background is basically just a straight line. It is often appropriate when there is no rise in the baseline across a peak envelope. The Shirley background is often appropriate when there is a rise in the baseline across the peak envelope. It is an empirical background. In general, the Shirley background at a given point is proportional to the number of counts in the peak envelope at higher kinetic energies. The Tougaard background is a longer energy range background.

However, mathematically, it is similar to the Shirley background in that it depends on the number of photoelectrons in the signal at higher kinetic energies than the point in question. However, at each point, the fraction of the peak envelope at higher kinetic energy is multiplied by a function that accounts for the attenuation of the electrons in the material. In other words, the Tougaard background takes into consideration the inelastic losses of the photoelectrons in the material under study. In addition, there are actually different types of Tougaard backgrounds with different numbers of parameters. Tougaard-type backgrounds enjoy a deeper theoretical foundation than the other backgrounds. However, they are also more challenging to apply because they require some knowledge of the material. In general, all three backgrounds give reasonable results when appropriately applied. However, the Shirley background usually underestimates the areas of peak envelopes, while the Tougaard background can overestimate them.

The types of synthetic peaks and background in an XPS peak fit should be reported. Poorly chosen synthetic peaks and backgrounds and/or unconstrained peaks[53] in XPS peak fits can lead to meaningless results. Figure 5 shows that both the peak shapes and backgrounds of XPS peak fits in the literature are minimally reported (only reported about 10% of the time). This lack of reporting makes it difficult to know whether they have been performed appropriately.

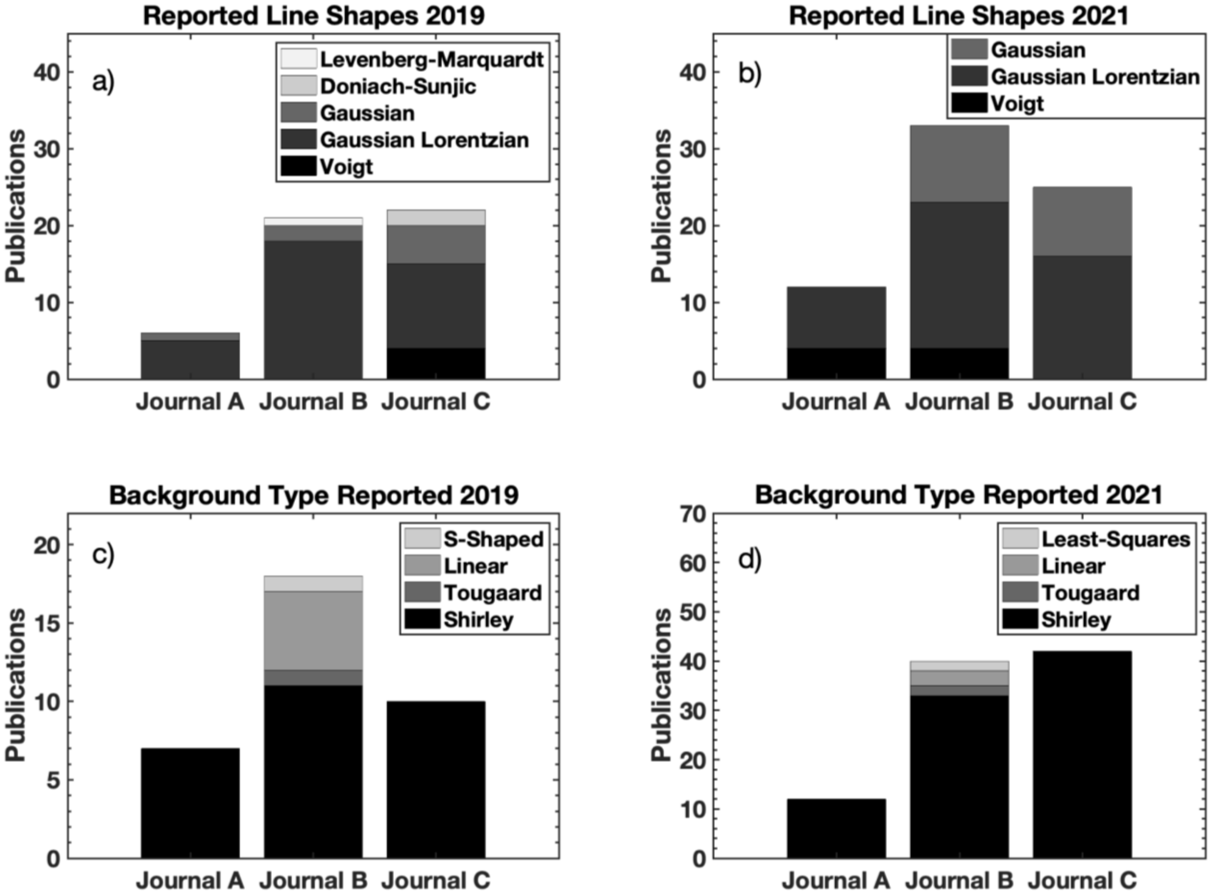

FIG 6. Data reporting from 2019 and 2021 from three journals showing (a) – (b) the types of synthetic peaks and (c) – (d) backgrounds used to fit XPS data. In (c) – (d), the 'Tougaard' backgrounds reported were 'U Poly Tougaard' backgrounds.

More specific information about the synthetic peak shapes and backgrounds used in peak fits in the literature is shown in Fig. 6. Figure 6(a) – (b) reveals that, in both 2019 and 2021, Gaussian-Lorentzian and Gaussian peak shapes were most widely used. Voigt peak shapes are less commonly employed. We believe some authors may have meant 'GLP' or 'GLS' when they reported that they used a Gaussian peak shape. As previously noted, the GLP and GLS peak shapes are adequate for many applications. In addition, Gaussians are also not a bad choice in some cases. Indeed, the commonly used GLP(30) peak shape, where the '30' refers, approximately, to the amount of Lorentzian character in the peak, is quite similar to a Gaussian.[46] Nevertheless, the Voigt peak shape better represents the underlying physics of photoemission, and we hope it will be used more widely in the future.

The Shirley background was the most widely used background in our survey of the literature (see Fig. 6(c) – (d)). Indeed, Journal B, which has a focus on surfaces, was the only journal in our survey that reported spectra fit with any other background. Nevertheless, some of the fits we surveyed appeared to have used linear backgrounds but failed to report it. This use of inferred linear backgrounds was not included in Fig. 6. In many cases, the Shirley background is a reasonable choice for a peak fit. As discussed previously, it is often appropriate when there is a rise in the baseline on the high binding energy side of a peak envelope. Even when there is no rise in the baseline across a peak envelope, the Shirley background often works well. In these cases, it will, in general, closely resemble a linear background, and peak areas (in reasonable fits) will be almost the same with either background. There are, however, situations where we believe a Tougaard background is a better choice. Shirley backgrounds can break down when applied to some transition metals. Furthermore, sometimes the Tougaard background better matches the baseline rise on the high binding energy side of peak envelopes. For example, the rise in the baseline at higher binding energy from some polymer samples is better modeled with a Universal Polymer Tougaard background than a Shirley background.[54-56] These universal polymer backgrounds are often preferable above other backgrounds because of their flexibility.

Modern XPS data analysis is only possible with advanced software designed for this purpose. Long gone are the days when a scientist might cut a printed peak out of a piece of paper with scissors and weigh the paper on a balance to determine its area. Thus, the software used for XPS data analysis should be reported. The various software packages used for XPS data analysis have different and sometimes unique capabilities. For example, CasaXPS[57] has peak shapes that are unique to it.[58-61] Figure 7(a) – (b) summarizes whether the peak fitting software was reported in the publications we surveyed. In both years, only about 15% of the publications reported the type of software used. Interestingly, Journal B (again the journal with the greatest emphasis on surfaces) had the largest percentage of publications reporting the data analysis software. Figure 7(c) – (d) summarizes the software packages that were reported in 2019 and 2021. In both years, CasaXPS was most widely reported/used.

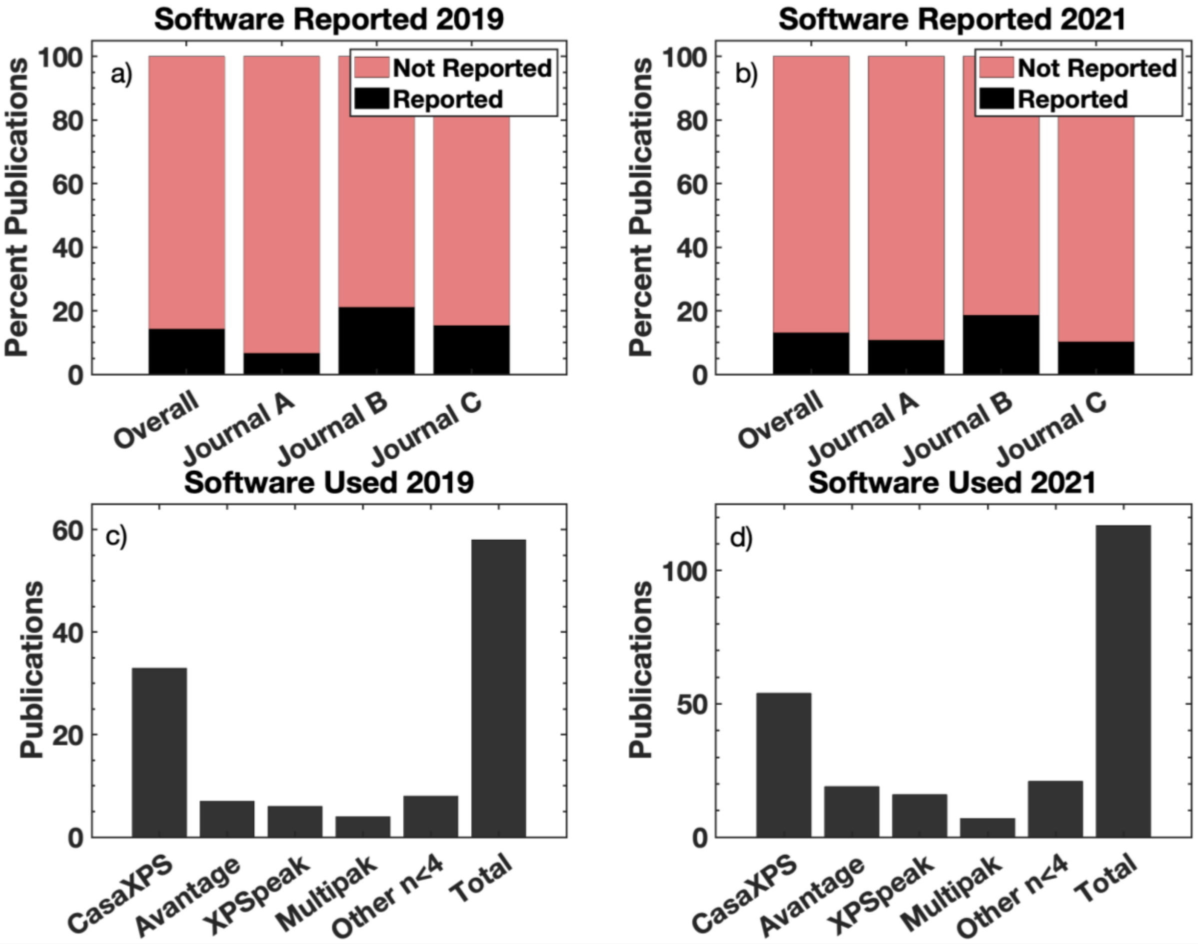

FIG 7. (a) – (b) XPS analysis software (black, lower bars) reported in publications in 2019 and 2021. In general, only ca. 15% of the publications surveyed reported the XPS analysis software that was used. (c) – (d) Different types of analysis software reported by number of publications.

CasaXPS was the most reported software in both years, followed by Avantage (from Thermo Fisher), then the open source XPSpeak, and then Multipak (from Physical Electronics).

D. Recommendations for reporting XPS results

The following information and parameters should be provided when XPS data and data analysis are reported. Not all these parameters were surveyed in our study. Nevertheless, the general lack of reporting found in this study suggests that all these parameters are underreported. While these lists are not exhaustive – a specialist will probably want more information, e.g., about the spectrometer, it should be adequate for most journals. The consistent reporting of these parameters would be a significant improvement over the level of reporting in the literature now.

Instrument/experimental parameters:

1. The manufacturer and model of the spectrometer.
2. The X-ray anode, or source and X-ray energy, e.g., Al Kα with 1486.6 eV, include beamline and synchrotron, if applicable.
3. Whether the source is monochromatized or non-monochromatized.
4. The type of energy analyzer, e.g., concentric hemispherical analyzer (CHA).
5. The X-ray power, beam size, and any beam rastering.
6. The geometry of the measurement, i.e., the angle between the sample plane and the entrance to the electron energy analyzer and the angle with the X-ray source. Alternatively, the system geometry may be described with respect to the sample normal.
7. The lens mode of the analyzer (wide angle, angle-resolved, focused spot).
8. The pressure during the analysis or the instrument base pressure.

9. The energy resolution (pass energy) of the survey and narrow scans and number of scans taken.
10. The dwell times or scan time and step sizes of the spectra.
11. Whether (and how) charge compensation was employed, and if the spectra were energy corrected, e.g., by referencing to the C 1s peak.
12. How the sample was mounted, e.g., whether it was electrically isolated from the instrument or grounded to it.
13. Anything else that is relevant or unique to the experiment, e.g., if the sample was tilted for angle resolved XPS, *in situ* preparation of the sample, the use of near-ambient pressure XPS, information about the polarization vector of the incident light, etc.
14. If a sample damage test was conducted.

If peak fitting was performed, the following parameters should be reported. These include:

1. The background selected.
2. The types of synthetic peaks used, e.g., Voigt, Gaussian, Lorentzian, or a pseudo-Voigt (GLS or GLP) functions.
3. Constraints placed on the synthetic peak shapes.
4. The software used for peak fitting.
5. The source of any sensitivity factors used, or the procedure used to determine them.

We recommend that published XPS spectra show the following information and/or contain the following formatting. Some of these recommendations only apply to fitted spectra.

1. In plotted spectra, binding energy should increase to the left, as per convention.
2. The raw data should be shown, not some processed, e.g., smoothed, form of it.
3. The background chosen should be shown, and it should be present under the entire peak envelope (over reasonable limits).
4. The sum of the synthetic peaks should be present, so that it can be compared to the raw data.
5. The individual, synthetic peaks in the fit should be shown, unless there is justification to combine them.[62, 63]
6. The residuals to the fit should be shown. This is an important, graphical way of assessing the quality of a fit. The residuals reveal where a fit matches and does not match the raw data.[53]

III. Summary and Conclusions

We have presented a survey of the literature that reveals inadequacies in reporting in the literature. Among papers with XPS results, ca. 30% do not report the spectrometer, ca. 60% do not report the X-ray source, and 80% or more do not report the pass energy, spot size, analysis software, peak shapes (in fitting), or background (in fitting). We believe this considerable lack of reporting has consequences. It makes it harder for a reader to assess the quality of any XPS data analysis, and therefore the quality of the work itself. It also makes it harder for the work to be reproduced. The current situation is unsatisfactory for both the assessment and replication of many results in the literature. It may be a symptom of the amount of poor quality XPS data in the literature. Improvements in reporting is the joint responsibility of authors, analysts, editors and reviewers. We believe the suggestions for reporting in this work can be rather easily followed.

Indeed, our lists are less comprehensive than current ISO standards[42] for XPS data reporting. Nevertheless, reporting at the level suggested in this work would be a substantial improvement over the current situation.

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AUTHOR DECARATIONS

Conflicts of Interest

The authors have no conflicts to disclose

DATA AVAILABILITY STATEMENT

The statistical results that support the findings of this study may be made available on request from the corresponding author. The specific evaluations of spectra that were made in this study will not be made available due to privacy and ethical restrictions.

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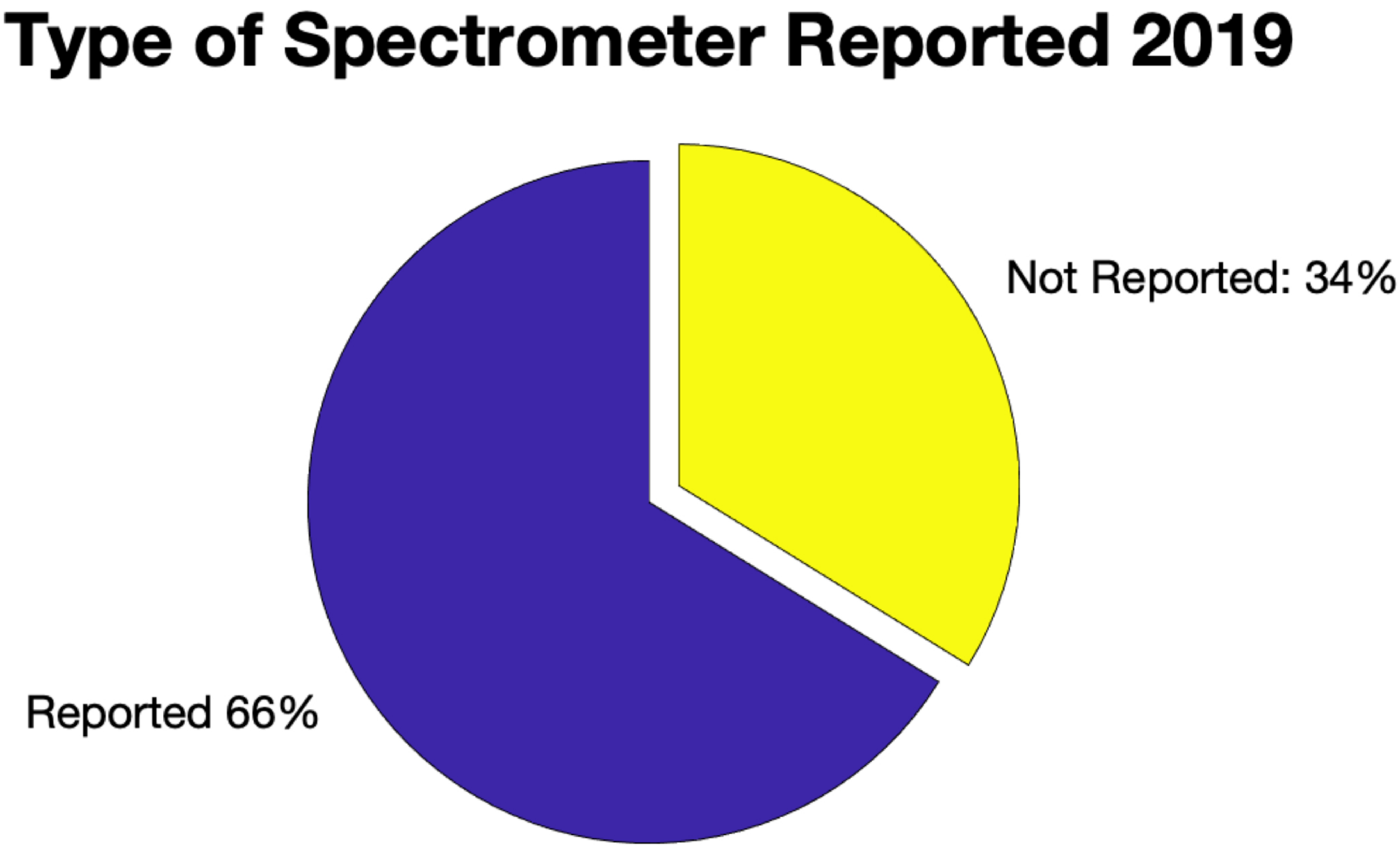
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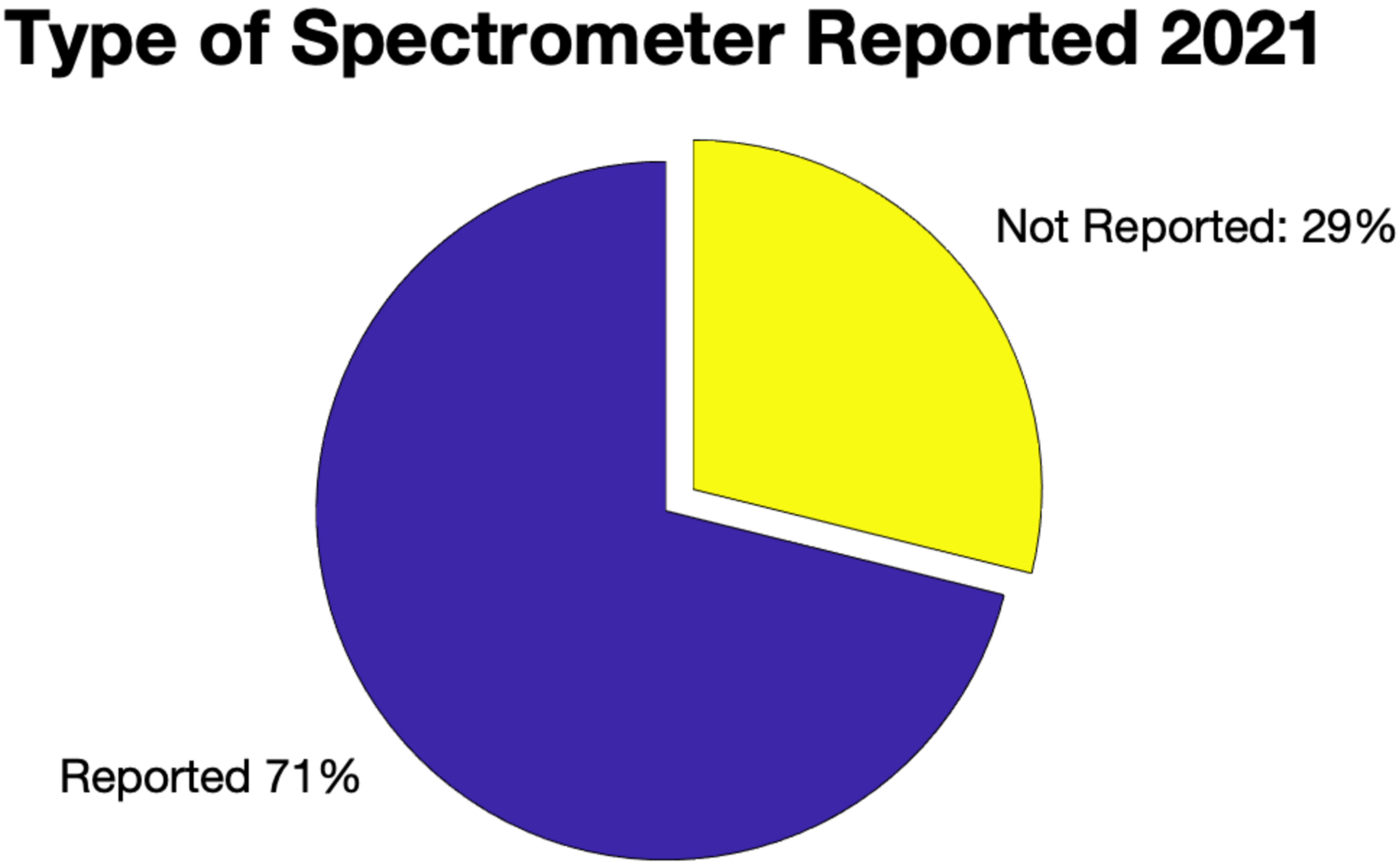
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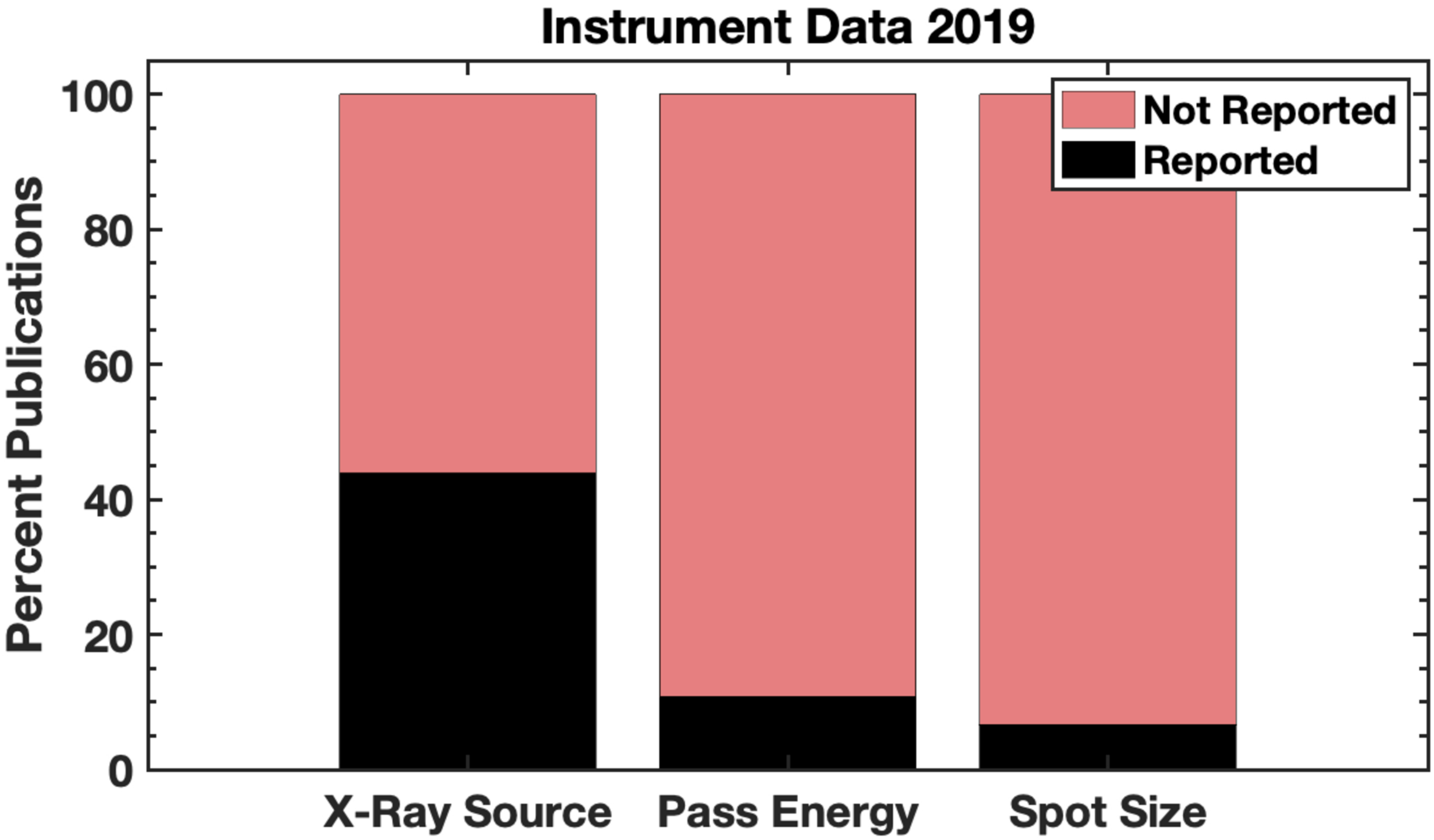

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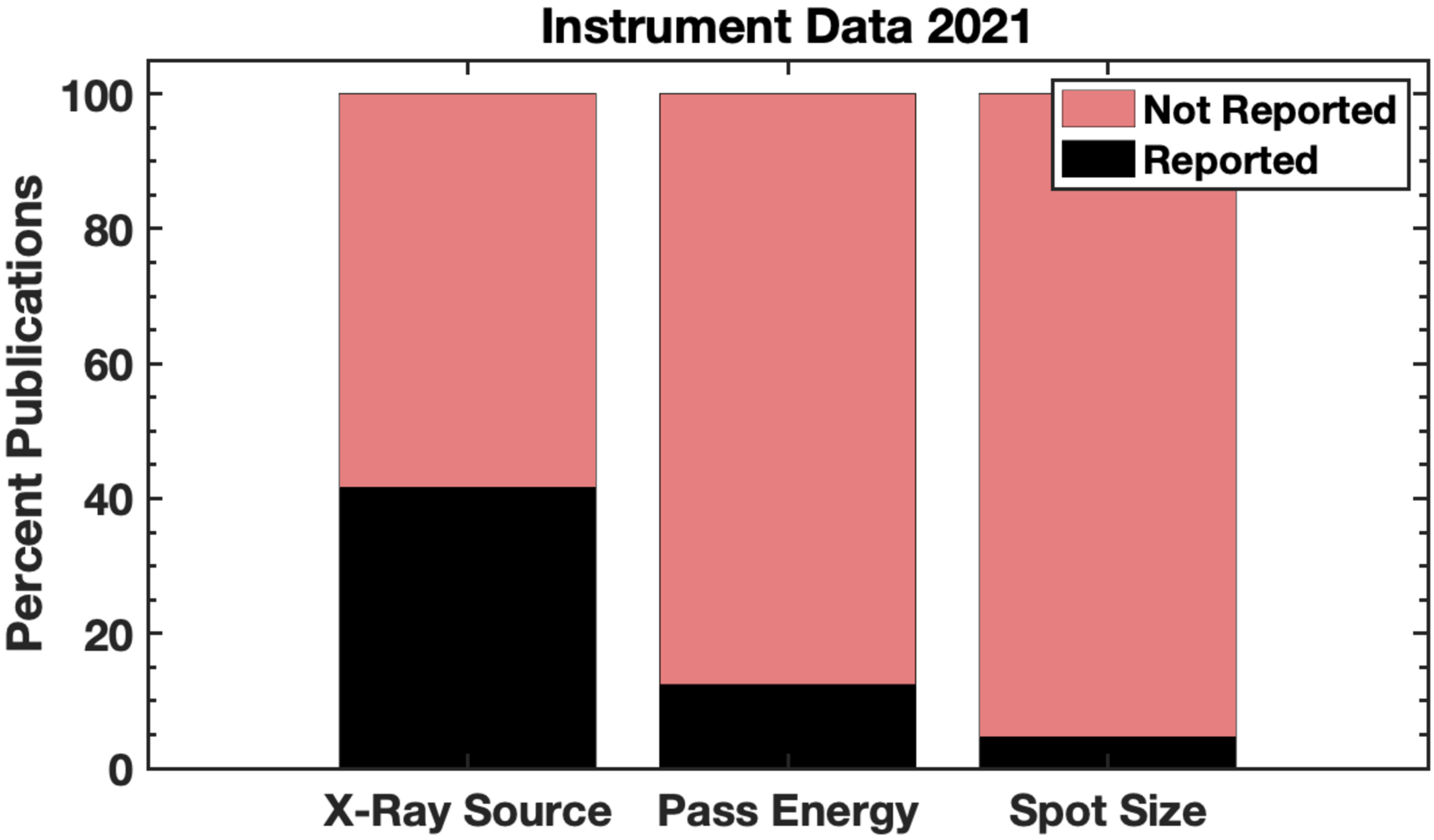

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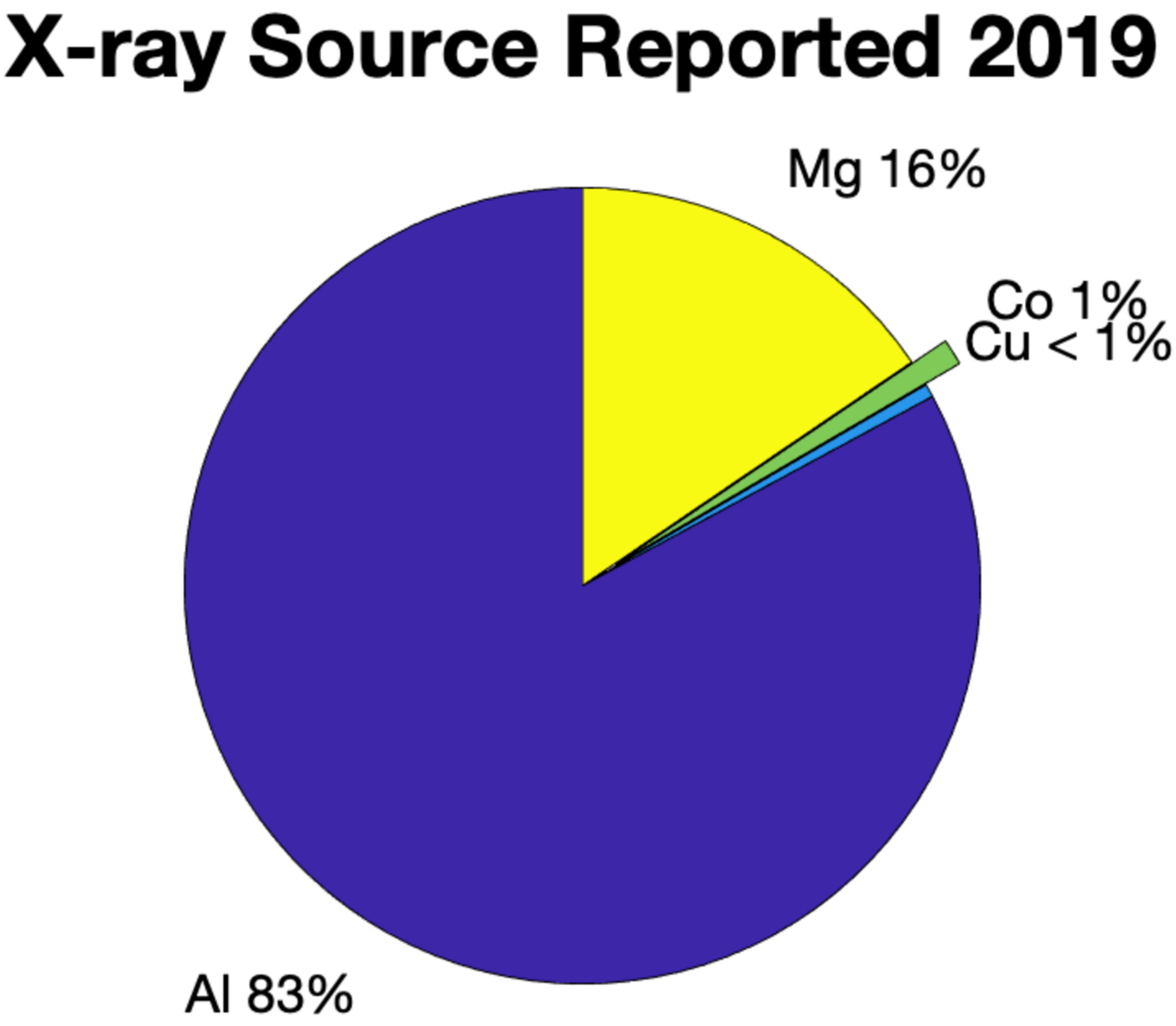
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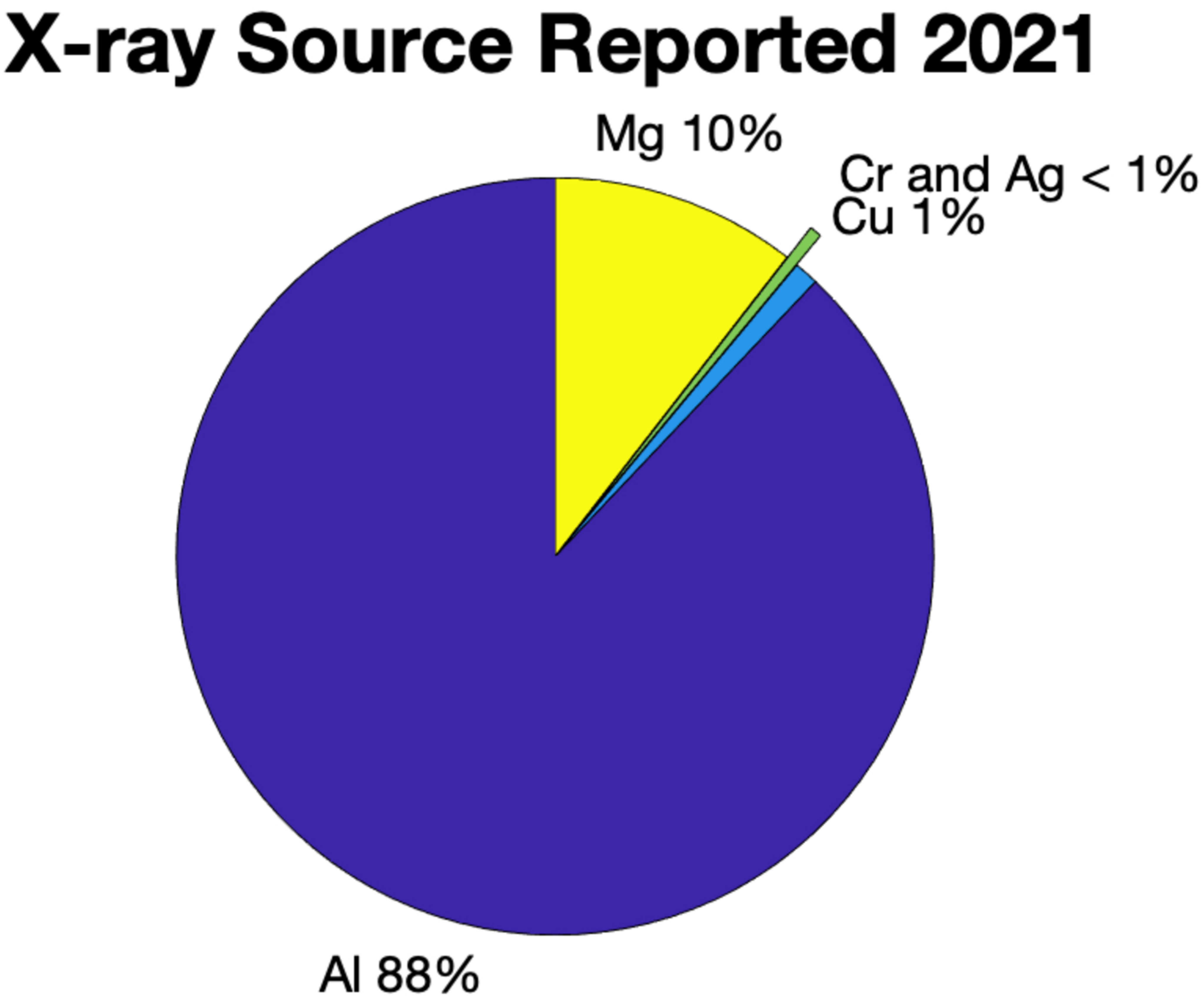
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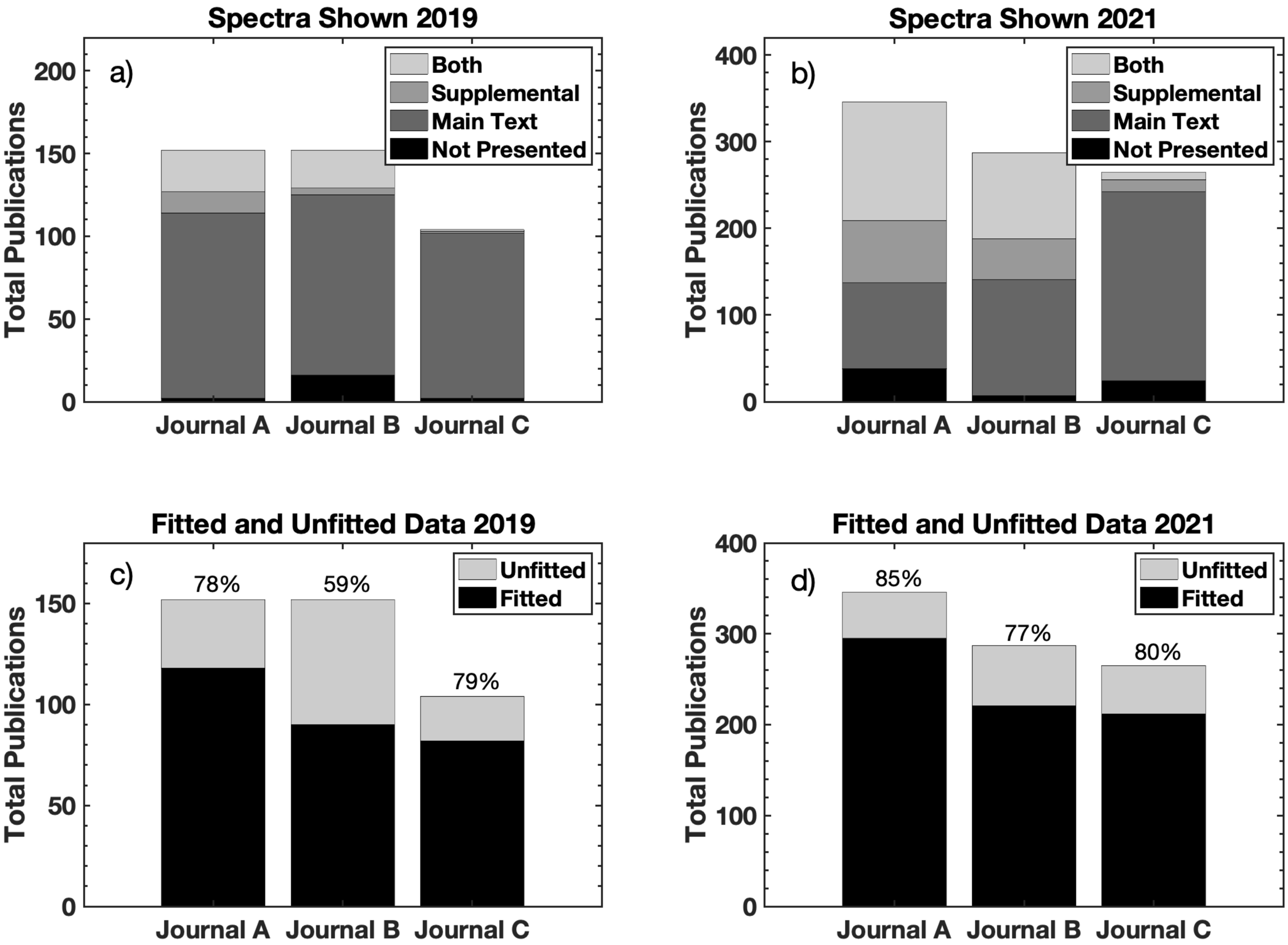
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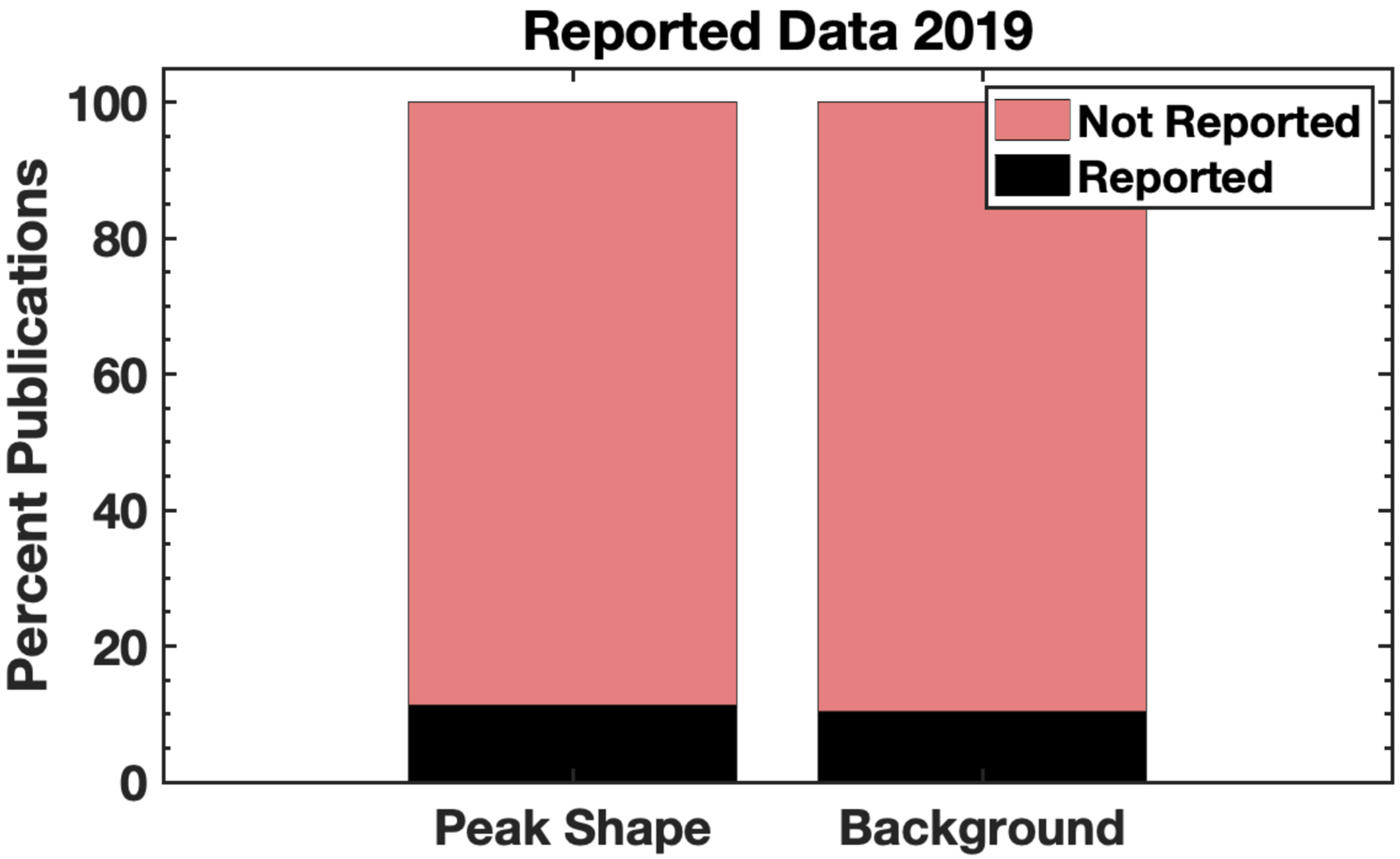
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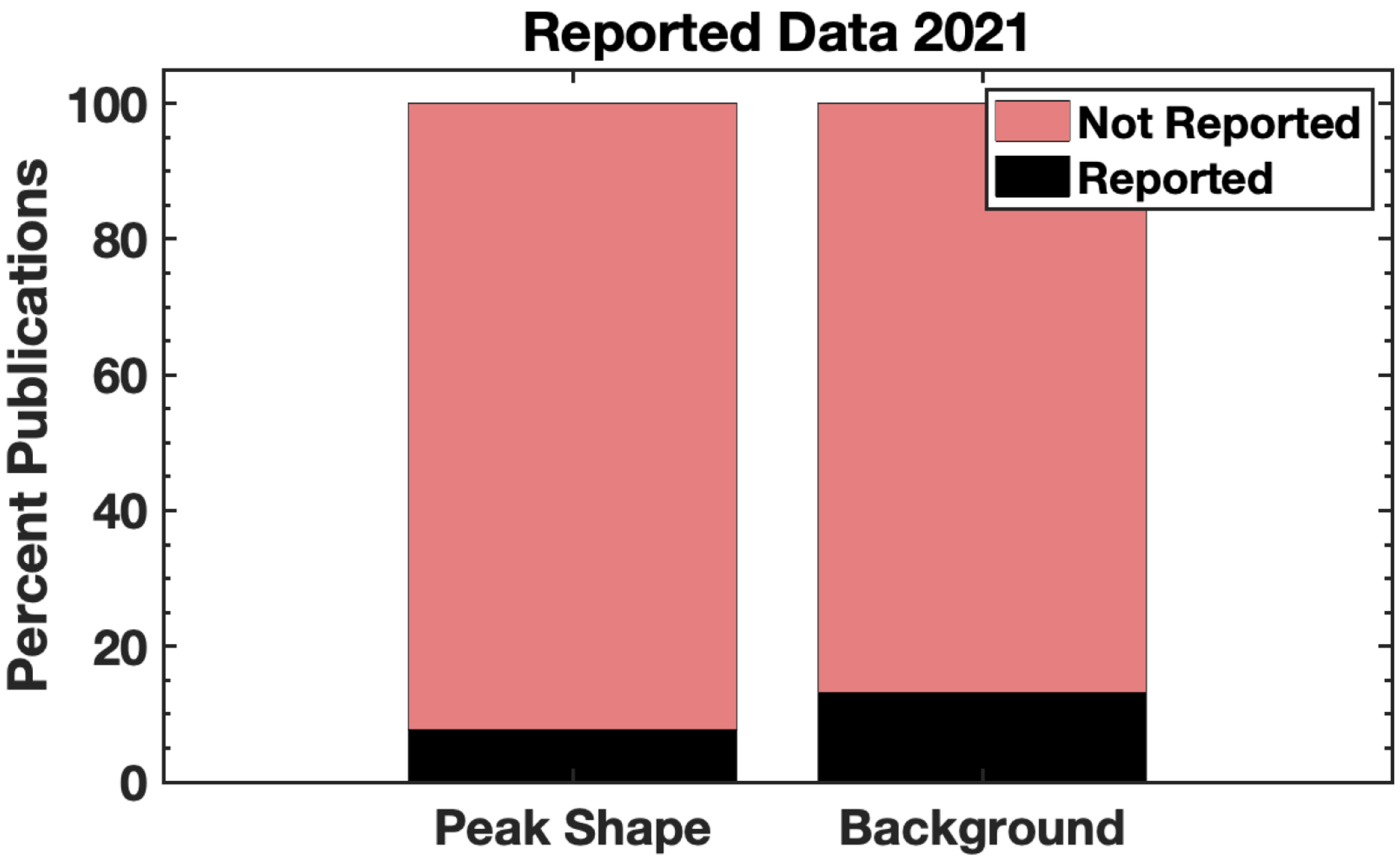
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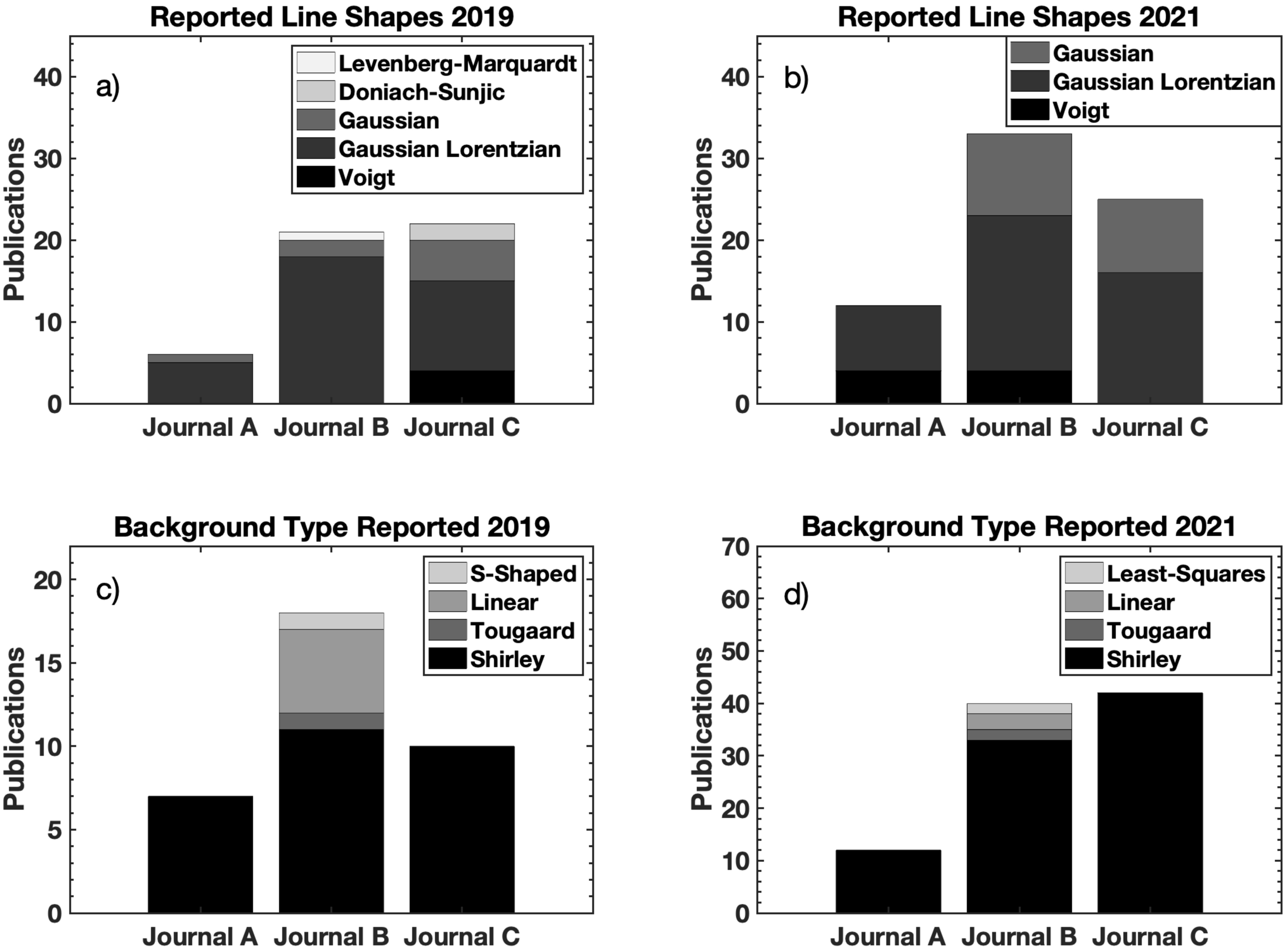
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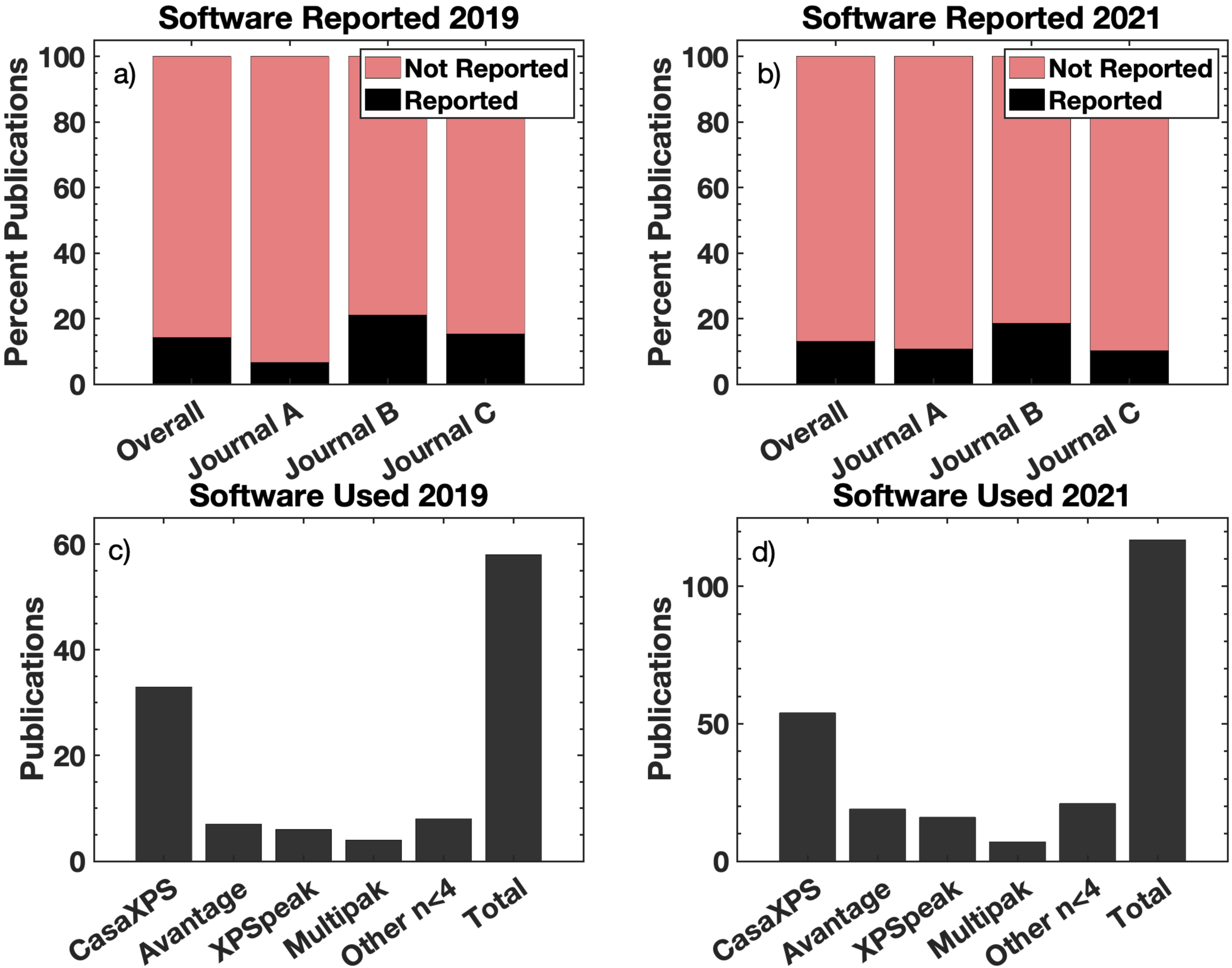
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